L4 ANSWER 1 OF 3 CAPLUS COPYRIGHT 2008 ACS on STN
AN 2002:345277 CAPLUS
ED Entered STN: 09 May 2002
TI Systematic arrangement structural optical material and its manufacturing method. [Machine Translation].
IN Honda, Takashi
PA [NAME NOT TRANSLATED], Japan
SO Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKXXAF
DT Patent
LA Japanese
IC ICM C30B030-06
ICS G02B001-02; G02B005-18; G02B005-28
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
PI	JP 2002128600	A	20020509	JP 2000-355016	20001018	<--
PRAI	JP 2000-355016		20001018			

CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
JP 2002128600	ICM	C30B030-06
	ICS	G02B001-02; G02B005-18; G02B005-28
	IPCI	C30B0030-06 [ICM,7]; C30B0030-00 [ICM,7,C*]; G02B0001-02 [ICS,7]; G02B0001-00 [ICS,7,C*]; G02B0005-18 [ICS,7]; G02B0005-28 [ICS,7]
	IPCR	G02B0005-28 [I,C*]; G02B0005-28 [I,A]; C30B0030-00 [I,C*]; C30B0030-06 [I,A]; G02B0001-00 [I,C*]; G02B0001-02 [I,A]; G02B0005-18 [I,C*]; G02B0005-18 [I,A]

AB [Machine Translation of Descriptors]. As for purpose of this invention, are the systematic arrangement structural optical material which the ultraviolet ray, the light and the light in the infrared ray territory Bragg is reflected and the times when the manufacturing method is offered. The systematic arrangement structural optical material of this invention designates the standard deviation of particulate grain size of the colloidal solution which it is stabilized as 20% or less of mean diameter, is produced in the false crystal which it occurs in that colloidal solution by growing false crystal to optional size including the vibration of acceleration below 10G above 0.1g or / and the stress vibration above 20Pa. This time, the same form as the crystal form which fine structure takes, or, two or more of said crystal form unites and growth of false crystal becomes easy the form which is brought together, or, by using the container of the form which possesses the crystal face of portion of said crystal form, also control of crystal orientation becomes possible. False crystal in the colloidal solution can control modulus of elasticity with ion concentration and macromolecule addition. In addition, also solidification is easy between the particulate of false crystal by filling up with the macromolecule and the inorganic material.

L4 ANSWER 2 OF 3 WPIX COPYRIGHT 2008 THOMSON REUTERS on STN
AN 2002-676930 [73] WPIX
DNC C2002-191071 [73]
DNN N2002-535116 [73]
TI Optical material for crystalline monochrometer, includes single crystal which is grown to specific size in colloidal solution and has controlled orientation along specific direction
DC L03; P81; U11; U12; V07
IN HONDA T
PA (HOND-I) HONDA T
CYC 1
PI JP 2002128600 A 20020509 (200273)* JA 8[0] <--
ADT JP 2002128600 A JP 2000-355016 20001018

PRAI JP 2000-355016 20001018
IPCR C30B0030-00 [I,C]; C30B0030-06 [I,A]; G02B0001-00 [I,C]; G02B0001-02 [I,A]; G02B0005-18 [I,A]; G02B0005-18 [I,C]; G02B0005-28 [I,A]; G02B0005-28 [I,C]
AB JP 2002128600 A UPAB: 20050903
NOVELTY - A single crystal is grown in a colloidal solution to a size of 1 cm along three mutually orthogonal directions. The orientation of the crystal is controlled along the required direction.
DETAILED DESCRIPTION - An INDEPENDENT CLAIM is included for optical material manufacturing method.
USE - Optical material used for crystalline monochrometer.
ADVANTAGE - The crystallinity of the optical material is improved and the strength of the optical material in narrow bandwidth is increased, hence the usage range of the optical material is extended.
MC CPI: L04-B01A
EPI: U11-A01; U12-A01; V07-F02B; V07-K05

L4 ANSWER 3 OF 3 JAPIO (C) 2008 JPO on STN
AN 2002-128600 JAPIO
TI ORDERED-STRUCTURE OPTICAL MATERIAL AND METHOD FOR PRODUCING THE SAME
IN HONDA TAKASHI
PA HONDA TAKASHI
PI JP 2002128600 A 20020509 Heisei
AI JP 2000-355016 (JP2000355016 Heisei) 20001018
PRAI JP 2000-355016 20001018
SO PATENT ABSTRACTS OF JAPAN (CD-ROM), Unexamined Applications, Vol. 2002
IC ICM C30B030-06
ICS G02B001-02; G02B005-18; G02B005-28
AB PROBLEM TO BE SOLVED: To provide an ordered-structure material which causes Bragg reflection of light in the region of ultraviolet rays, visible light, and infrared rays, and provide a method for producing the same.
SOLUTION: The ordered-structure optical material is produced as follows: the standard deviation of particle diameters of fine grains in a stabilized colloidal solution is controlled to be <=20% of an average particle diameter, and a resultant quasicrystal in the colloidal solution is grown to an arbitrary size by adding to the quasicrystal a vibration at >=0.1 G and <=10 G acceleration and/or a stress vibration of >=20 Pa. At this time, the quasicrystal can be easily grown and its crystal orientation can be easily controlled by using a vessel which has the same shape as a crystal form of a close-packed structure, a shape formed by combining two or more of the crystal forms, or a shape of a partial crystal face of the crystal form. The elasticity of the quasicrystal in the colloidal solution can be controlled with ion concentration or addition of high polymers. The solidification of the quasicrystal also can be easily performed by filling the gaps among fine particles of the quasicrystal with high polymers or an inorganic material.

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PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2002-128600

(43)Date of publication of application : 09.05.2002

(51)Int.Cl. C30B 30/06
G02B 1/02
G02B 5/18
G02B 5/28

(21)Application number : 2000-355016

(22)Date of filing : 18.10.2000

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(72)Inventor : HONDA TAKASHI

(54) ORDERED-STRUCTURE OPTICAL MATERIAL AND METHOD FOR PRODUCING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an ordered-structure material which causes Bragg reflection of light in the region of ultraviolet rays, visible light, and infrared rays, and provide a method for producing the same.

SOLUTION: The ordered-structure optical material is produced as follows: the standard deviation of particle diameters of fine grains in a stabilized colloidal solution is controlled to be ≤20% of an average particle diameter, and a resultant quasicrystal in the colloidal solution is grown to an arbitrary size by adding to the quasicrystal a vibration at ≥0.1 G and ≤10 G acceleration and/or a stress vibration of ≥20 Pa. At this time, the quasicrystal can be easily grown and its crystal orientation can be easily controlled by using a vessel which has the same shape as a crystal form of a close-packed structure, a shape formed by combining two or more of the crystal forms, or a shape of a partial crystal face of the crystal form. The elasticity of the quasicrystal in the colloidal solution can be controlled with ion concentration or addition of high polymers. The solidification of the quasicrystal also can be easily performed by filling the gaps among fine particles of the quasicrystal with high polymers or an inorganic material.

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1]Regular arrangement structure light study material characterized by ****** including a false crystal grown up into a size of 1 cm or more by a 2-way at least among three directions which are mutually right-angled and cross while controlling crystal orientation of a false crystal produced in a colloidal solution in the direction of the purpose.
[Claim 2]The regular arrangement structure light study material according to claim 1, wherein standard deviation of particle diameter of particles which constitute a false crystal produced in a colloidal solution is 20% or less of mean particle diameter.
[Claim 3]A manufacturing method of the regular arrangement structure light study material according to claim 1 adding vibration of acceleration not more than more than 0.1G10G, or/and stress vibration of not less than 20 Pa to a false crystal produced in a colloidal solution, and making false crystal growth perform.
[Claim 4]The same shape as a crystal form which the close-packed structure takes in a container used when growing up a false crystal produced in a colloidal solution, Or a manufacturing method of the regular arrangement structure light study material according to claim 1 growing up a false crystal which controlled crystal orientation by using a container of shape where two or more of these crystal forms combined, or shape which has some crystal faces of this crystal form.
[Claim 5]The regular arrangement structure light study material according to claim 1 considering it as a solvent which ion concentration used as water below $6x10^{-2}$mol/l, or/and an organic solvent a solution which exists among particles of particles which constitute a false crystal grown up in a colloidal solution, or was made to dissolve a polymer material in this solution.
[Claim 6]The regular arrangement structure light study material according to claim 1 filling up with a polymer material or an inorganic material between particles of particles which constitute

a false crystal grown up in a colloidal solution.

[Translation done.]

* NOTICES *

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention is an optical material in which regular arrangement structure is shown a cycle comparable as the wavelength of light, and relates to the regular arrangement structure light study material which can start Bragg reflection in ultraviolet, infrared rays, and a light region, and its manufacturing method.
[0002]
[Description of the Prior Art]Conventionally, although the crystalline monochrometer for X-rays which used single crystals, such as silicon, is known by the optical material using Bragg reflection, only X-rays can carry out Bragg reflection to it, but it has a fault as which reflected wave length is specified by the kind of crystal. Although the diffraction grating using diffraction, the interference filter using interference of light, etc. are known as a monochrome-ized element near the light range, the manufacture of a diffraction grating is complicated and there is a fault to which a diffraction pattern becomes dark. An interference filter also has a fault which creation says that it is difficult and it difficult to change arbitrarily the wavelength which can carry out [monochrome]-izing.
[0003]The opal is known as a natural mineral which starts Bragg reflection. Even if large, the color unevenness of the colored spot which shows a false crystal structure in an opal is also intense at about several millimeters, and its direction is also irregular, and it is not used other than jewelry. Although the opal Mr. diffraction luminescence film which made the colloidal solution of the particles of micron order adhere to a substrate, and made JP,8-234007,A produce the Bragg reflection like an opal is indicated, The three-dimensional structure with strong Bragg reflection intensity was not acquired, but was homogeneous, and difficult to obtain the diffraction luminescence film of a large area.
[0004]The colloid crystal is known as what starts Bragg reflection with a colloidal solution. The

false crystal which the homogeneous silica particle and polystyrene particles of the nano order carried out the closest packing to the colloid crystal, and resembled the crystal well is formed. Since the lattice spacing of the bottom of a case is hundreds of nm wholly as a lattice point, the Bragg reflection in a light region happens, the intervals, i.e., the particles, of a lap of this particle, and the play of color like an opal is shown. [of an array surface] In a colloid crystal, what has the same direction of a crystal axis is especially called colloid single crystal in every portion. For example, the particulate dispersion liquid which assumes the reflet like an opal is indicated by JP,6-100432,A. Although the gazette explains that the size of a colloid single crystal changes with the size of the particles of a colloidal solution, particle size distribution, particle concentration, contamination ion concentration, the particle surface density of electric charge, etc. to 0.1-10 mm, The appearance of a colloid single crystal takes place irregularly, and control of crystal orientation is not made, either.

[0005]

[Problem(s) to be Solved by the Invention]The purpose of this invention is to provide the regular arrangement structure light study material which carries out Bragg reflection of the light in ultraviolet rays, visible light, and an infrared area, and its manufacturing method.

[0006]

[Means for Solving the Problem]If a large-sized colloid single crystal can be created easily, this invention person To achieve the above objects, ultraviolet rays, It is thought that it is applicable as a spectrum in visible light and an infrared area, and a monochrome-ized optical material, As a result of inquiring wholeheartedly, standard deviation of particle particle diameter of a colloidal solution is made into 20% or less of mean particle diameter, A colloid crystal is produced, after making below into $6x10^{-3}$mol/l concentration (it is described as ion concentration below) which set a positive ion and negative ion in a colloidal solution and stabilizing it, It found out that a colloid crystal was grown up into arbitrary sizes, and large-sized colloid single crystal-ization could be performed by adding vibration of acceleration not more than more than 0.1G10G, or/and stress vibration of not less than 20 Pa to the colloid crystal. At this time, growth of a colloid single crystal found out that became easy and control of crystal orientation was also attained by using a container which has some crystal faces of the same shape as a crystal form which the close-packed structure takes, or this crystal form. Although wavelength which carries out Bragg reflection with an angle which looks at a colloid single crystal changes, since this colloid single crystal also has character as an elastic body, a Bragg reflection wavelength can also be changed according to external force. By filling up between particles of particles which constitute a colloid single crystal with inorganic materials, such as polymer materials, such as an acrylic, and glass, it found out that it could create easily also as a solid optical material, and this invention was completed.

[0007]Hereafter, details of this invention are explained. The colloidal solution concerning this

invention can use one kind of one kind or two kinds or more of mixed solutions, water or an organic solvent, or water, and an organic solvent, or two kinds or more of mixed solutions as dispersion liquid. Particulate dispersion Metal oxide particles, such as silica, a titania, alumina, zirconia, and copper I oxide, Or organic matter particles, such as metal particles, such as metal hydroxide particles, such as a ferric hydroxide, or selenium, and nickel, or polystyrene, polyethylene, methacrylic resin, and synthetic latex, can be used.

[0008]A method stable by adding sodium hydroxide, sodium oxide, etc. and making the pH of a colloidal solution into the alkalinity of 8-11, a method of reducing ion concentration of a colloidal solution, etc. are used for a method of stabilizing so that particles in a colloidal solution generally may not be condensed. A method of making ion concentration of a colloidal solution below $6x10^{-3}$mol/L with cation exchange resin and anion exchange resin or a method of making ion concentration of a colloidal solution below $6x10^{-3}$mol/L by dialysis can be used for stabilization of a colloidal solution concerning this invention.

[0009]Methods of making a colloid crystal generating include closest packing arrangement-ization by natural sedimentation of particles. Although crystal growth progressed toward the upper part according to sedimentation of particles and this method became an aggregate of a needlelike colloid single crystal which is 1-3 mm in width, it was difficult to enlarge width of a needlelike colloid single crystal.

[0010]Another methods of making a colloid crystal generate include a method of removing ion of a colloidal solution to a degree very much. If ion is removed, it is known that an electric double layer currently formed in the particle surface will become thick. Since particles serve as particles of a size including an electric double layer, if a volume ratio which particles including an electric double layer occupy becomes close to 74.04% of a packing index which the closest packing shows, a colloid crystal will generate them. However, by this method, even if large, only an about 8-mm colloid single crystal was obtained, but crystal orientation was also irregular.

[0011]this invention person vibration of acceleration not more than more than 0.1G10G to a colloid crystal by which it was generated in a colloidal solution continuously, Or by adding intermittently, a colloid single crystal grew and it found out that a colloid single crystal with a size of 1 cm or more could be formed by a 2-way at least among three directions which are mutually right-angled and cross. $g=Ax(2pif)^2x10^{-4}$ and (g have acceleration cm/sec^2, A has amplitude mum, and there is a relation of pitch Hz) to acceleration of vibration to add of f. Growth will become slow if growth of a colloid single crystal is quick when acceleration is large, and acceleration is small. However, it turned out that growth of a colloid single crystal will not take place if acceleration is less than [0.1G], but a colloid crystal will be destroyed if acceleration is larger than 10G.

[0012]Stress vibration may be sufficient as vibration to add. Particles in a colloidal solution form an electric double layer in the surface, and this electric double layer carries out a role of an elastic body. If stress is applied to a colloidal solution from the exterior, vibration can be given by an electric double layer's pushing, and contracting and changing stress to apply. When width of a stress change added continuously or intermittently stress vibration of not less than 20 Pa to a colloid crystal, a colloid single crystal grew and it found out that a colloid single crystal with a size of 1 cm or more could be formed by a 2-way at least among three directions which are mutually right-angled and cross. Growth became slow when growth of a colloid single crystal was quick when width of a stress change was large, and width of a stress change was small. However, when width of a stress change was smaller than 20 Pa, growth of a colloid single crystal did not take place. Same result was obtained even if it added simultaneously vibration of acceleration not more than more than 0.1G10G, and stress vibration with a stress change width of not less than 20 Pa. By adding vibration with a moderate colloid crystal, a phenomenon in which growth takes place resembles well a phenomenon in which an atom causes a thermal oscillation and grain growth happens, when the usual polycrystal heat-treats near the melting point.

[0013]The shape as a crystal form which the close-packed structure takes where it is the same when adding vibration in this invention, Or growth of a colloid single crystal found out that became easy and control of crystal orientation was also attained by using a container of shape where these two or more crystal forms combined, or shape which has some crystal faces of this crystal form. When the bottom analyzed a Bravais lattice at the time for particles of a colloid single crystal from visible light Bragg reflection wholly as a lattice point, it became clear that a face-centered cubic lattice was usually taken and a simple hexagonal lattice was taken by conditions. A tetrahedron, hexahedron, octahedron, a method dodecahedron of slanting, three-side 34 face pieces, neighborhood 34 face piece, five-side 34 face pieces, hextetrahedron, three-side 38 face pieces, neighborhood 38 face piece, five-side 38 face pieces, hexoctahedron, tetrahexahedron, dihexahedron, a double dodecahedron, etc. are among crystal forms which a face-centered cubic lattice takes fundamentally. A roppo cylindrical-surface object, a double roppo cylindrical-surface object, double dihexahedron, double trigonal bipyramid, a double roppo cone, a ** square face piece, dihexahedron, trigonal bipyramid, a roppo cone, etc. are among crystal forms which a simple hexagonal lattice takes. Shape with a crystal habit is also contained in these crystal forms. That is, not only as for a regular hexahedron but a rectangular parallelepiped to which only a specific surface became large, hexahedron is contained.

[0014]Crystal orientation of a colloid single crystal grown up with these containers becomes in the almost same direction as a crystal face which the same crystal form as shape of a container to be used has. That is, if a crystal face is expressed with a Miller index, in the case

of a hexahedron-shaped container, a field equivalent (100) to a field appears in each field, when it is a method dodecahedron [of slanting]-shaped container, each field will turn into a field (110) and an equivalent field, and, in the case of an octahedron-shaped container, it will become a field where each field is equivalent to a field (111). In the case of a roppo cylindrical-surface bodily-shape-like container, a field where the cylindrical surface of a field (1010) or the bottom is equivalent to a field (0001) is taken. However, when ** adds a vibration strong only against the direction of a specific surface in the case of a container which enlarged only a specific surface extremely, a colloid single crystal may grow so that the field may turn into a field (111).

[0015]A light source and a telescope were attached to a spherical coordinate measuring device, and a decision of a Miller index of a crystal face of a colloid single crystal was made by measuring coordinates of a crystal face and a glancing angle of Bragg reflection which start Bragg reflection. A field predicted that a face-centered cubic lattice has a characteristic extinction rule, and can observe Bragg reflection, By (111), (200), (220), (311), (222), (400), (331), (420), and (422), if reflectivity sets reflectivity of a field (111) to 100, 100, 47, 22, 24, 7, 2, 8, 8, and 8 will be predicted in order. About (200) with strong reflectivity and comparatively low (111) indices of crystal plane, and (220), if the appearance direction of a crystal face is expressed with a spherical coordinate (phi, rho), As for (111), (90degree00', 90degree00'), and (220) become (45degree00', 90degree00') (45degree00', 54degree44'), and (200). In lattice spacing d_{hkl} of a crystal face (hkl), it is $d_{hkl}^2=a^2/(h^2+k^2+l^2)$ (a is a grating constant and hkl is a Miller index).

There is ****** and it is $2d_{hkl}$sintheta=nlambda (as for a glancing angle and lambda, in theta, wavelength and n are positive integers) in Bragg reflection.

There is ******. A Miller index was determined from these relations and crystal face coordinates.

[0016]A container of shape where two or more of crystal forms which the close-packed structure takes combined is a container of shape where a regular-octahedron crystal face of an equilateral triangle combined in a corner of a regular hexahedron, etc., for example. A container which has some crystal faces of a crystal form which the close-packed structure takes is a container, a container of the fourth page of a lower part of octahedron, a container of the second page of a lower part of a tetrahedron, etc. which removed only the upper surface of hexahedron, for example, and other fields may not be, when a colloidal solution does not fall, although a curved surface may be sufficient. It becomes easy to produce distortion etc. in a colloid single crystal, and crystal completeness falls as the number of crystal faces which a container has decreases. When using this container, a direction vertical to a crystal face which this container has of the direction of vibration to add is good.

[0017]Particles in a colloidal solution used for this invention require that standard deviation of particle diameter should use 20% or less of mean particle diameter. If standard deviation of particle diameter is larger than 20% of mean particle diameter, when particles carry out the closest packing, arrangement nonuniformity will be made, and crystal growth will be checked remarkably. If standard deviation of particle diameter is larger than 30% of mean particle diameter, the generation of a colloid crystal itself will be checked remarkably. Dispersion in particle diameter makes a colloid single crystal produce imperfection. Since disorder equivalent to fluctuation of a lattice defect and a partial crystal axis which are produced into a common crystal also to a colloid single crystal arises, in order to fully exhibit a function as a spectrum or a monochrome-ized optical material, 10% or less of mean particle diameter of standard deviation of particle diameter is preferred.

[0018]A colloid single crystal used for this invention has the character as an elastic body. If ion concentration of a colloidal solution is made below into $6x10^{-3}$mol/l, particles can stabilize for it and hold an electric double layer on the surface also in a colloid single crystal, and this electric double layer will carry out a role of an elastic body. If stress is applied to a colloid single crystal from the exterior, an electric double layer pushes and contracts, in the bottom, lattice spacing of a case will be wholly shortened as a lattice point, and change will produce on wavelength of light which carries out Bragg reflection, intervals, i.e., particles, of a lap of particles in a colloid single crystal. [of an array surface] When an elastic modulus of a colloid single crystal was measured from change of wavelength of light which carried out Bragg reflection to stress using an expression of relations of the aforementioned Bragg reflection, about 10^{2} - 10^{3}Pa were shown. When polymer materials, such as polyvinyl alcohol, are dissolved to a colloidal solution, an elastic modulus improves and about 10^{3} - 10^{9}Pa are shown.

[0019]It became possible to change wavelength of Bragg reflection light, without destroying a false crystal structure, even if it applied stress to a colloid single crystal by making into 20% or less of mean particle diameter standard deviation of particle diameter of particles in a colloidal solution used for this invention. If it is not less than 20% of mean particle diameter about standard deviation of particle diameter of particles, a gap of a lattice defect and a crystal axis will arise by applying stress. When it was not less than 30%, it checked that a false crystal structure broke gradually by applying stress.

[0020]

[Example]Hereafter, an example is given and this invention is explained concretely. The laser type size distribution measuring device performed measurement of the mean particle diameter of the particles of the colloidal solution of this invention, and standard deviation. The distortion of the lattice defect of a colloid crystal and a crystal axis was checked with the stereoscopic microscope and the spectrometer. Growth of the colloid single crystal was checked by a

stereoscopic microscope and viewing. Ion chromatography performed ion concentration measurement.

[0021]The pure water 453g and the 25% ammonia solution 68g were mixed to 1586 g of example 1 ethanol, and it agitated at 0 **. Agitation mixing was performed for 1 hour, mixing the solution which diluted 25.76 g of alt.silicic-acid tetraethyl with 183.08 g of ethanol, and fixing reaction temperature at 0 ** in the place which became constant [temperature]. Then, ethanol and ammonia were removed in the evaporator and the colloidal solution which performs the inspissation by centrifugation and contains a silica particle 60% of the weight was obtained. Put this colloidal solution into the inside dimension method 2cmx2cmx2cm container made from a hexahedron acrylic, settled it, particles were made to sediment automatically, and the colloid crystal was made to generate. Then, when 1 Hz of vibration was added with the acceleration 1G and the colloid single crystal was grown up, the colloid single crystal single to the limit of a container grew. The mean particle diameter of this colloid particle was 291 nm, and standard deviation was 5% of mean particle diameter. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0022]The reaction condition which performs agitation mixing for 1 hour fixing example 2 reaction temperature at 0 **, Except having replaced with the reaction condition which performs agitation mixing for 1 hour while raising reaction temperature at 1-hour a rate of 3 ** from -1.5 **, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. The mean particle diameter of this colloid particle was 293 nm, and standard deviation was 7% of mean particle diameter. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0023]The reaction condition which performs agitation mixing for 1 hour fixing example 3 reaction temperature at 0 **, Except having replaced with the reaction condition which performs agitation mixing for 1 hour while raising reaction temperature at 1-hour a rate of 6 ** from -3 **, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. The mean particle diameter of this colloid particle was 294 nm, and standard deviation was 20.5% of mean particle diameter. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0024]With the example 4 acceleration 1G, except having replaced 1 Hz of vibration with 5-Hz vibration with the acceleration 10G, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0025]With the example 5 acceleration 1G, except having replaced 1 Hz of vibration with 1 Hz of vibration with the acceleration 10G, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.
[0026]With the example 6 acceleration 1G, except having replaced 1 Hz of vibration with 1 Hz of vibration with the acceleration 5G, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.
[0027]With the example 7 acceleration 1G, except having replaced 1 Hz of vibration with 0.5 Hz of vibration with the acceleration 1G, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.
[0028]With the example 8 acceleration 1G, except having replaced 1 Hz of vibration with 1 Hz of vibration with the acceleration 0.1G, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.
[0029]The colloidal solution which contains example 9 silica particle 60% of the weight was created in the same procedure as the procedure of example 1 statement. Put into the inside dimension method 2cmx2cmx2cm container made from a hexahedron acrylic which provided the feed port of the diameter of 6 mm for applying stress to the upper part for this colloidal solution, settled, particles were made to sediment automatically, and the colloid crystal was made to generate. Then, when 20-Pa stress vibration was added with the cycle of 1 Hz and the colloid single crystal was grown up from the feed port of the diameter of 6 mm, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.
[0030]With the cycle of 101 Hz of examples, except having replaced 20-Pa stress vibration with 200-Pa stress vibration with the cycle of 1 Hz, when the colloid single crystal was grown up like Example 9, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.
[0031]With the cycle of 111 Hz of examples, except having replaced 20-Pa stress vibration with 2000-Pa stress vibration with the cycle of 1 Hz, when the colloid single crystal was grown

up like Example 9, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0032]With the cycle of 121 Hz of examples, except having replaced 20-Pa stress vibration with 20000-Pa stress vibration with the cycle of 1 Hz, when the colloid single crystal was grown up like Example 9, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0033]With the cycle of 131 Hz of examples, except having replaced 20-Pa stress vibration with 200000-Pa stress vibration with the cycle of 1 Hz, when the colloid single crystal was grown up like Example 9, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0034]The colloidal solution which contains example 14 silica particle 60% of the weight was created in the same procedure as the procedure of example 1 statement. Put into the inside dimension method 2cmx2cmx2cm container made from a hexahedron acrylic which provided the feed port of the diameter of 6 mm for applying stress to the upper part for this colloidal solution, settled, particles were made to sediment automatically, and the colloid crystal was made to generate. Then, while adding 200-Pa stress vibration with the cycle of 1 Hz from the feed port of the diameter of 6 mm, when it added 1 Hz of vibration with the acceleration 1G and the colloid single crystal was grown up, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0035]Except the inside dimension method having replaced the example 15 inside-dimension method 2cmx2cmx2cm container made from a hexahedron acrylic with one-side the container made from a regular-octahedron acrylic which is 2 cm, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (111) which measured the indices of crystal plane of the colloid single crystal in regular-octahedron container each field.

[0036]Except having replaced the example 16 inside-dimension method 2cmx2cmx2cm container made from a hexahedron acrylic with the inside dimension method 0.3cmx4cmx10cm container made from a hexahedron acrylic, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It just (111) measured the indices of crystal plane of the colloid single crystal in the largest field (4 cm x 10 cm) with the hexahedron container. Even if the shape of a container is hexahedron, this is considered for the field where particle density is the highest (111) to grow preferentially when a specific field is extremely large.

[0037]The example 17 inside-dimension method 2cmx2cmx2cm container made from a hexahedron acrylic, Except having replaced with the container made from an acrylic with which the triangular regular-octahedron crystal face whose one side is 5 mm combined in the corner of the 2cmx2cmx2cm regular hexahedron, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. ******** with indices of crystal plane of the colloid single crystal in the field belonging to a regular octahedron equivalent to (111) in respect of being equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in the field belonging to a regular hexahedron.

[0038]Except having replaced the inspissation by example 18 centrifugation with the inspissation by centrifugation, after adding ethanol so that content may be 50% of the weight, when the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0039]The inspissation by example 19 centrifugation except having replaced ethanol with the inspissation by centrifugation, after content added benzene 40% of the weight so that content might be 40% of the weight, When the colloid single crystal was grown up like Example 1, the colloid single crystal single to the limit of a container grew. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0040]The colloidal solution which contains example 20 silica particle 60% of the weight was created in the same procedure as the procedure of example 1 statement. After adding sodium chloride so that it may become the concentration of 1.44×10^{-5} mol/l to this colloidal solution, Put into the inside dimension method 2cmx2cmx2cm container made from a hexahedron acrylic which established the feed port of the diameter of 6 mm for applying stress in the upper part, settled, particles were made to sediment automatically, and the colloid crystal was made to generate. Then, 1 Hz of vibration was added with the acceleration 1G, and the colloid single crystal was grown up. In the place where the colloid single crystal became a size which is 2cmx2cmx2cm, when stress of 50 Pa was applied and change of the wavelength of the Bragg reflection light of a field (220) was checked by 70 degrees of glancing angles, it changed to 500 nm from 550 nm. From Bragg's equation, d_{220} was changing to 266 nm from 293 nm, and the elastic modulus was able to be found with 5.6×10^{2} Pa from this.

[0041]Except having replaced the concentration of example 211.44x10^{-5} mol/l with the concentration of 3×10^{-3} mol/l, after growing up a colloid single crystal like Example 20, when the elastic modulus of the colloid single crystal was measured, it was able to be found with 1.1×10^{3} Pa.

[0042]The pure water 453g and the 25% ammonia solution 68g were mixed to 1586 g of example 22 ethanol, and it agitated at 0 **. Agitation mixing was performed for 1 hour, mixing the solution which diluted 25.76 g of alt.silicic-acid tetraethyl with 183.08 g of ethanol, and fixing reaction temperature at 0 ** in the place which became constant [temperature]. Then, ethanol and ammonia were removed in the evaporator and it was referred to as 60 cc, and after dissolving the polyvinyl alcohol 2g, the colloidal solution which performs the inspissation by centrifugation and contains a silica particle 60% of the weight was obtained. Put this colloidal solution into the inside dimension method 2cmx2cmx2cm container made from a hexahedron acrylic with the 60-cc funnel for sample introduction, settled it on the upper part, particles were made to sediment automatically, and the colloid crystal was made to generate. Then, 1 Hz of vibration was added with the acceleration 1G, and the colloid single crystal was grown up. It was set to 1.1×10^{4}Pa when the elastic modulus of this colloid single crystal was measured.
[0043]Example 23 -- except having replaced the elastic modulus with measurement, after measurement evaporated [the elastic modulus of this colloid single crystal] 35 cc of supernatant liquid of this colloid single crystal, when the colloid single crystal was grown up like Example 22 and the elastic modulus was measured, it was set to 5×10^{7}Pa.
[0044]After drying the colloid single crystal created in example 24 Example 1, it heat-treated at 800 ** for 1 hour. The methyl methacrylate monomer was ****(ed) after cooling to the room temperature, it irradiated with ultraviolet rays, methyl methacrylate was stiffened, and regular arrangement structure light study material was obtained.
[0045]After drying the colloid single crystal created in example 25 Example 1, it heat-treated at 800 ** for 1 hour. The pressure-medium container made from carbon was filled up with borosilicate glass powder with a softening temperature of 470 ** after cooling to the room temperature, and the heat treatment body was put into the center. Hotpress processing was carried out for this by 550 ** and 2×10^{7}Pa for 2 hours, and the heat treatment body was impregnated with borosilicate glass. Then, unnecessary borosilicate glass was shaved off and regular arrangement structure light study material was obtained.
[0046]Except having replaced the example 26 inside-dimension method 2cmx2cmx2cm container made from a hexahedron acrylic with the glass beakers whose diameter at the bottom is 4 cm, When the colloid single crystal was grown up like Example 1, the colloid single crystal grew selectively and the colloid single crystal of the size length and whose width are 1.5 cm appeared. It just (111) measured the indices of crystal plane of the colloid single crystal in the bottom of a beaker. This is considered for the field where particle density is the highest (111) to grow preferentially over the bottom which is a flat surface.
[0047]Creation operation of the colloidal solution which contains the silica particle of example

27 example 1 statement 60% of the weight was repeated 10 times. Put this colloidal solution into the inside dimension method 5cmx5cmx5cm container made from a hexahedron acrylic, settled it, particles were made to sediment automatically, and the colloid crystal was made to generate. Then, when 1 Hz of vibration was added with the acceleration 1G and the colloid single crystal was grown up, the colloid single crystal single to the limit of a container grew, and it turned out that growing gigantic is easy. It was a field equivalent to the place (200) which measured the indices of crystal plane of the colloid single crystal in hexahedron container each field.

[0048]The reaction condition which performs agitation mixing for 1 hour fixing comparative example 1 reaction temperature at 0 **, When a colloid single crystal is grown up like Example 1 except having replaced with the reaction condition which performs agitation mixing for 1 hour, raising reaction temperature at 1-hour a rate of 8 ** from -4 **, growth of a colloid single crystal is seen selectively, but. It was mutually right-angled and the growth to the size of 1 cm or more was not seen by a 2-way at least among three crossing directions. In the colloid single crystal, the fluctuation of a crystal axis had arisen selectively. The mean particle diameter of this colloid particle was 295 nm, and standard deviation was 23.5% of mean particle diameter.

[0049]While the reaction condition which performs agitation mixing for 1 hour fixing comparative example 2 reaction temperature at 0 ** raised reaction temperature at 1-hour a rate of 10 ** from -5 **, grew up the colloid single crystal like Example 1 except having replaced with the reaction condition which performs agitation mixing for 1 hour, but. Growth was hardly seen but play of color became indistinct selectively conversely. The mean particle diameter of this colloid particle was 295 nm, and standard deviation was 31% of mean particle diameter.

[0050]Although the colloid single crystal was grown up like Example 1 with the comparative example 3 acceleration 1G except having replaced 1 Hz of vibration with 1 Hz of vibration with the acceleration 0.05G, growth was hardly seen.

[0051]Although the colloid single crystal was grown up like Example 1 with the comparative example 4 acceleration 1G except having replaced 1 Hz of vibration with 1 Hz of vibration with the acceleration 12G, growth was hardly seen, but play of color became indistinct selectively conversely, and the colloid crystal collapsed selectively.

[0052]With the comparative example 5 acceleration 1G, except having replaced 1 Hz of vibration with 10-kHz vibration with the acceleration 50G, when the colloid single crystal was grown up like Example 1, the colloid crystal collapsed.

[0053]Although the colloid single crystal was grown up like Example 9 with the cycle of 61 Hz of comparative examples except having replaced 20-Pa stress vibration with 15-Pa stress vibration with the cycle of 1 Hz, growth was hardly seen.

[0054]Although the colloid single crystal was grown up like Example 9 with the cycle of 71 Hz

of comparative examples except having replaced 20-Pa stress vibration with 10-Pa stress vibration with the cycle of 1 Hz, growth was hardly seen.
[0055]Although the colloid single crystal was grown up like Example 1 except the inside dimension method having replaced the comparative example 8 inside-dimension method 2cmx2cmx2cm container made from a hexahedron acrylic with the container made from a spherical acrylic which is 3 cm in diameter, it grew up to be only a size of about 8 mm.
[0056]Although the colloid single crystal was grown up like Example 20 except having replaced the concentration of comparative example 91.44×10^{-5}mol/l with the concentration of 3.5×10^{-3}mol/l, particles condensed and crystal growth did not happen.
[0057]
[Effect of the Invention]Since it can be made to grow up to be an as desired size and the crystal orientation of a colloid single crystal can also be controlled, the colloid single crystal of this invention is applicable as the spectrum in ultraviolet rays, visible light, and an infrared area, and a monochrome-ized optical material. Although a Bragg reflection wavelength can be changed according to a glancing angle, the character as an elastic body of a colloid single crystal is applied, and since a Bragg reflection wavelength can also be changed by applying external force, Bragg reflection of the wavelength of the wider range can be carried out in one crystal face. A new regular arrangement structure light study material is obtained by filling up with polymers or an inorganic material after drying a colloid single crystal body.
[0058]A colloid single crystal has acceleration insufficient for growing up a crystal, when vibration not more than 0.1G and stress vibration are 20 Pa or less, but vibration of the acceleration not more than more than 0.1G10G and stress vibration of not less than 20 Pa are the best for growth of a colloid single crystal. However, in case of vibration of the acceleration beyond 10G, it will be too strong and a colloid single crystal will collapse. When the standard deviation of particle diameter makes the particles in a colloidal solution 20% or less of mean particle diameter, a good colloid single crystal is obtained. If the standard deviation of particle diameter is larger than 20% of mean particle diameter, when particles carry out the closest packing, arrangement nonuniformity will be made, and crystal growth will be checked remarkably. If the standard deviation of particle diameter is larger than 30% of mean particle diameter, the generation of a colloid crystal itself will be checked remarkably. Since an electric double layer cannot obtain sufficient thickness but the condensation between particles takes place in case of more than 6×10^{-3}mol/l, the ion concentration of a colloidal solution is considered that a colloidal solution and a colloid single crystal become unstable. Although the elastic modulus of a colloid single crystal changes also with ion concentration, a high elastic modulus can be obtained by dissolving polymer materials, such as polyvinyl alcohol.
[0059]The container of a colloid single crystal will be penetrated to 175 nm - 3.4 micrometers in

case of quartz to the wavelength of 150 nm - 5 micrometers, if sapphire is used, and although a dispersed solution also shows absorption strong against the wavelength 3.3 micrometers and near 6 micrometer, as for water, it shows transmissivity sufficient on the other wavelength. Since absorption in an infrared area can use as a dispersed solution what fully removed ion with little liquid paraffin, hexachlorobutadiene, etc., the spectrum of the light in ultraviolet rays, visible light, and an infrared area and monochrome-ization are attained.

[0060]Acrylic resins used as a packing material after drying a colloid single crystal light from 250 nm Through, Although it has absorption 1.18 micrometers and near 1.4 micrometer, it is transparent in the wide wavelength range, and the spectrum of light and monochrome-ization also of low melting glass and the regular arrangement structure light study material solidified in the wide wavelength range since it was transparent are attained in the wide wavelength range. Since the solidified regular arrangement structure light study material can carry out [****]-izing, as an application, application is possible also as curving mold monochromators, such as the Johansson type, besides a monotonous monochromator, monochromatic light with strong intensity with narrow spectral band width can be acquired, and an application range is dramatically wide.

[0061]Therefore, the regular arrangement structure light study material obtained from this colloid single crystal is the optimal as the spectrum in ultraviolet rays, visible light, and an infrared area, and a monochrome-ized optical material. Since color changes with the directions to see, this regular arrangement structure light study material has highly a dramatically promising effect of which it complains to human being's aesthetic sense also as accessories. The value as science teaching materials for understanding a crystal structure and Bragg reflection is high.

[Translation done.]